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PAPER

CRIMINALISTICS

Andria H. Mehltretter,[1] M.S.; Maureen J. Bradley,[1] Ph.D.; and Diana M. Wright,[1] Ph.D.

Analysis and Discrimination of Electrical Tapes: Part II. Backings*,†

ABSTRACT: The backings of 90 black electrical tapes were analyzed to evaluate the chemical components of these films, the ability of individual techniques to discriminate samples, and the ability of the techniques combined to distinguish samples. The techniques utilized and their respective discrimination results were stereomicroscopy and physical measurements, to include observation of surface features of the backing, width, and thickness measurements (*c.* 64%); Fourier transform infrared spectroscopy (FTIR) using a microscope accessory (*c.* 83%); pyrolysis–gas chromatography/mass spectrometry (Py-GC/MS; *c.* 81%); and scanning electron microscopy/energy-dispersive spectroscopy (SEM/EDS; *c.* 87%). Ninety-four percent of the backings were discriminated through this combination of analytical methods. Finally, evaluating these results in conjunction with previously published data on the analysis of the adhesives from the same set of electrical tapes provided an overall discrimination of nearly 96%.

KEYWORDS: forensic science, trace evidence, electrical tape, backing, discrimination, stereomicroscopy, Fourier transform infrared spectroscopy, pyrolysis–gas chromatography/mass spectrometry, scanning electron microscopy/energy-dispersive spectroscopy

Electrical tapes are common evidentiary specimens submitted to forensic laboratories with a request for comparative analysis. Various analytical techniques can be applied, but assessing the significance of a failure-to-discriminate result is complicated. In an attempt for some clarification, the FBI Laboratory's Chemistry Unit embarked on a study to evaluate the discrimination of black electrical tapes using standard methods of analysis.

The most obvious physical characteristic noted for electrical tapes is the color of the backing. Black is the most common color, but others are manufactured and sold. Even among black tapes, the degree of sheen can range from a dull, matte finish to a high gloss finish. Upon closer inspection, the surface features and textures vary and are likely imparted during the manufacturing process. Keto found that six brands of electrical tapes could be distinguished based on the surface textures of the backing (1). Although a range of widths are available, approximately 19 mm is common. Similarly, thicknesses can vary and has been cited as the only difference between different products from the same manufacturer (2).

In addition to the various physical characteristics that can be observed and measured for electrical tapes, the chemical composition of a tape can also be evaluated. Adhesive composition was discussed previously (3). The backing generally consists of a polymer, plasticizers, fillers, pigments, flame retardants, stabilizers, and lubricants (4). The polymer of most electrical tapes is polyvinyl chloride (PVC), but other materials (e.g., polyester and polyimide) can be used (5). As untreated PVC polymer film is rigid and unstable, plasticizers are used to soften the PVC. Examples include compounds of the following classes: phthalates, sebacates, and phosphates (4), but analyses at the authors' laboratory demonstrate that adipates and trimellitates are also used. Because the plasticizer can migrate out of the backing as well as into the adhesive, a barrier coating, such as polymethylacrylate, may be applied (6). Fillers are added to reduce manufacturing costs (4) and might include titanium dioxide, calcium carbonate, barium sulfate, kaolin, and talc (1). PVC does not burn; however, the plasticizers in PVC may sustain burning of the backing. Therefore, flame retardants are added and can include organic halogen or phosphorus compounds, antimony trioxide, aluminum hydroxide, or magnesium carbonate (6). Stabilizers are used to prevent decomposition and help prevent degradation by ultraviolet irradiation (4) and could include lead carbonate; lead sulfate; stearates of calcium, lead, cadmium, and barium; dibutyl tin dilaurate; or diphenyl urea (1). All of these additives are not included in all electrical tapes or may not be present above the detection limits of frequently used analytical techniques (7).

Most chemical analyses of electrical tapes involve at least two of the following: infrared spectroscopy (IR), energy-dispersive spectroscopy (EDS), and pyrolysis in conjunction with either gas chromatography (GC) or mass spectrometry (8). A combination of chemical analysis techniques allows identification of the various chemical components of electrical tapes: EDS for the inorganic components, various pyrolysis techniques for the organic components, and IR for inorganic and organic components.

Within the FBI Laboratory, submitted samples are first evaluated by visual and microscopical means to assess physical characteristics such as adhesive and backing colors, degree of sheen, surface texture and features, width, and backing thickness. Overall tape

[1]Laboratory Division, Federal Bureau of Investigation, 2501 Investigation Parkway, Quantico, VA 22135.

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thicknesses are also measured when the adhesive is not obviously contaminated or degraded or when the tape is not obviously stretched or deformed. Tape ends are examined for possible end matches, but regardless of the presence of an end match, physically indistinguishable tapes are further evaluated for chemical composition and comparison. Current FBI protocol calls first for chemical analysis via Fourier transform infrared spectroscopy (FTIR) with a microscope attachment, followed by scanning electron microscopy (SEM) with EDS, and pyrolysis–gas chromatography/mass spectrometry (Py-GC/MS).

Three studies have recently been carried out regarding the forensic analysis of electrical tape samples. In the first, the backings of 67 rolls of electrical tape were analyzed by SEM to image the surface texture and also to evaluate the elemental composition (2). Differences were readily apparent in surface texture (roughness, calendering marks, and filler particle size) and elemental ratios. Microtexture and elemental composition were quite reproducible, even over long periods of time, and SEM was found to be highly reliable and discriminating for electrical tape analysis.

In the second study, the organic composition of the backings and the adhesives was evaluated by attenuated total reflectance FTIR (9). Seventy-two rolls were evaluated, and the accuracy of the data was even better than of the previous elemental analyses, with the adhesive component being even more discriminating than that of the backing.

The discrimination power of various techniques in the analysis of 90 electrical tape adhesives was recently reported by the current authors (3). It was found that stereomicroscopy could discriminate 53% of the comparison pairs. For each analytical technique, within-sample and within-group reproducibility was demonstrated. FTIR analysis yielded a discrimination of 67%, and Py-GC/MS analysis increased that discrimination to 83%: differences between groups were most frequently because of identification of the plasticizer(s) within the adhesive. Scanning electron microscopy/energy-dispersive spectroscopy (SEM/EDS) was the least discriminating technique at 17%, a result not surprising as the adhesives were not expected to contain inorganic components. Combining the results from all the techniques resulted in an overall discrimination of over 85%.

The second portion of this study, the analysis of electrical tape backings, is the subject of this publication. The purpose of each study was the same: to assess the range of physical characteristics and chemical compositions of electrical tapes, to evaluate the ability of individual techniques to discriminate samples, and to determine the discrimination obtained from using a combination of techniques. Finally, the results from analysis of both the adhesives and backings were compiled to determine the overall discrimination of electrical tape samples.

Materials and Methods

Tape Collection

This study involved the analysis of 90 black electrical tape samples utilizing current FBI Laboratory protocols. Most of the tapes were purchased by FBI personnel at discount stores or home-improvement retailers, marketed as general purpose or economy grade, and originated from Taiwan, China, or the U.S. Therefore, the sample set represented tapes that could be easily obtained by consumers and would be comparable to evidence submitted to forensic laboratories. Table 1 is a summary of the products that were evaluated in this work. For a number of samples, the manufacturer was not listed on the packaging. Further, it is common practice for tapes to be purchased from a manufacturer and distributed under one or more private labels. While Underwriters Laboratories (UL) numbers can be used to determine a tape manufacturer, they were not available for all tapes in the collection. As manufacturer information was not available for all samples, nor was the sample set selected to be representative of all manufacturers, the sample set is not intended to be used to provide sourcing information regarding a tape sample of unknown origin.

Microscopical Examinations and Physical Measurements

Physical characteristics of the backings were recorded following visual and stereomicroscopical evaluation. Using a ruler, the width was measured three times to the nearest 0.5 mm. For thickness measurements, samples are placed between the two faces of a digital micrometer. For thickness measurements of the backing alone, the adhesive was first removed with hexane. A minimum of five areas were measured, and the values were recorded to the nearest 0.05 mil (1 mil = 1/1000 inch). For such measurements, a significant difference on a pristine tape (not stretched, deformed, or highly contaminated) is generally considered to be a width difference greater than 1.0 mm or a thickness difference greater than 0.2 mil. These values are based on manufacturer tolerances.

FTIR

All samples were taken as thin peels and compressed between the two diamond windows of a compression cell (Thermo Scientific, Waltham, MA). They were then analyzed on a single diamond window using a Continuum microscope attached to a Nicolet Nexus 670 FTIR E.S.P. spectrometer with a MCT/A detector (4000–650 cm^{-1}) at a resolution of 4 cm^{-1} (Thermo Nicolet, Madison, WI). The aperture was roughly 100 × 100 μm, and the number of scans was 128. Although many samples were analyzed once, replicate analyses were conducted on numerous samples to confirm reproducibility of the data.

Py-GC/MS

All samples were taken as thin peels, weighed to approximately 60 μg, and placed in a quartz pyrolysis tube using quartz wool as a support medium to position the sample approximately 15 mm from the top. Pyrolysis was conducted using either an AS-2500 or AS-5250 CDS Analytical pyrolysis autosampler (Oxford, PA). The initial temperature was set at 300°C for 1 sec, ramped at a rate of 20°C/msec to 880°C, and then held at that temperature for 10 sec. The temperature of the interface was 321°C. The pyrolysis unit was coupled to an Agilent 6890 Gas Chromatograph (Agilent Technologies, Wilmington, DE). The GC column was a DB5-MS, 30 m, 0.25 mm inner diameter, with a 0.25-μm film thickness. The carrier gas was helium and had a purity of 99.99%. The GC was operated at an initial temperature of 50°C for 2 min, ramped at a rate of 13°C/min to 325°C, and held for 5 min. The GC inlet was operated at 300°C in 50:1 split mode with a split flow of 34.9 mL/min. The mass spectrometer was a single quadrupole Agilent 5973 Mass Selective Detector with a dedicated electron impact ionization source. The transfer line temperature was set to 300°C, and the source temperature was 230°C. Full scan mode was employed with a scan range of 34–650 m/z. Although many samples were analyzed once, replicate analyses were conducted on numerous samples to confirm reproducibility of the data.

TABLE 1—*Product information for sample set.*

Sample Roll	Brand Name	Product	UL	CSA Reference	Country of Origin
1	Marcy Enterprises, Inc.	MA 750	111K		Taiwan
2	Advance®	AT7, BS3924, 31/90Tp			England
3	Work Saver™, a Royal Tools brand	Stock no. 55, 5 color P.V.C Tape Assortment			China
4	Tesa Tape, Inc.	40201, No. 111 E52811A	362K		Taiwan
5	Tape It, Inc.	E-60	119K		Taiwan
6	Qualpack®	1346, 6-Color			China
7	Marcy Enterprises, Inc.	MA 750	111K		Taiwan
8	Manco®	200 MPH, AE-66	590J	LR31971	Taiwan
9	Archer® (Packaged for Radio Shack)	64-2349	590J		Taiwan
10	3M Scotch™	Super 88, 054007-06143	539H	LR48769	U.S.A.
11	3M Scotch™	Super 33+, 10414 NA	539H	LR48769	U.S.A.
12	3M Scotch™	Super 33+, 10455 NA	539H	LR48769	U.S.A.
13	3M Scotch™	Super 33+	539H	LR48769	U.S.A.
14	Frost King®	ET60	206T		Taiwan
15	3M Scotch™	Super 33+, 10455 NA	539H	LR48769	U.S.A.
16	3M	Tartan™ 1710, part no. 054007 49656	539H	LR48769	U.S.A.
17	3M Scotch™	Super 88 054007-06143	539H	LR48769	U.S.A.
18	3M Scotch™	Super 33+, Cat. 195NA	539H	LR48769	U.S.A.
19	3M Scotch™	Super 33+, Cat. 194NA	539H	LR48769	U.S.A.
20	3M Scotch™	Super 33+, 10414 NA	539H	LR48769	U.S.A.
21	Manco®	P-66	590J	LR31971	Taiwan
22	Manco®	667 Pro Series™	590J	LR31971	Taiwan
23	3M Scotch™	Super 88, 054007-06143	539H	LR48769	U.S.A.
24	3M Scotch™	Super 88, 054007-06143	539H	LR48769	U.S.A.
25	3M Scotch™	Super 33+, 054007-06132	539H	LR48769	U.S.A.
26	3M Scotch™	Super 33+, 054007-06132	539H	LR48769	U.S.A.
27	3M	Tartan™ 1710, part no. 054007 49656	539H	LR48769	U.S.A.
28	3M	Tartan™ 1710, part no. 054007 49656	539H	LR48769	U.S.A.
29	3M	Temflex™, 1700, 54007-69764	539H	LR48769	U.S.A.
30	3M	Temflex™, 1700, 54007-69764	539H	LR48769	U.S.A.
31	Regal®	Model ET-6			Taiwan
32	GE	GE2472-3DD	206T		Taiwan
33	3M Scotch™	Cat. 190			U.S.A.
34	3M	Tartan™ 1710, part no. 54007-49656	539H	LR48769	U.S.A.
35	Frost King®	ET60	206T		Taiwan
36	3M	Tartan™ 1710, part no. 49656	539H		U.S.A.
37	National	All-Purpose Grade	206T		Taiwan
38	Manco®	P-660	590J	LR31971	Taiwan
39	3M Scotch™	Super 33+, 3744NA	539H	LR48769	U.S.A.
40	3M	Tartan™ 1710, part no. 054007 49656	539H	LR48769	U.S.A.
41	3M Scotch™	Super 33+, 200NA	539H	LR48769	U.S.A.
42	National	All-Purpose	362K		Taiwan
43	3M	Tartan™ 1710, part no. 054007 49656	539H	LR48769	U.S.A.
44	3M	Tartan™ 1710, part no. 054007 49656	539H	LR48769	U.S.A.
45	Calterm®	49605	590J		Taiwan
46	Manco®	P-20	590J	LR31971	Taiwan
47	3M	Tartan™ 1710, part no. 054007 49656	539H	LR48769	U.S.A.
48	Tape-It	36-T			U.S.A.
49	Tape-It	36-T			U.S.A.
50	General Electric	GE2472-31D	206T		Taiwan
51	National	No. 101, E52811A	362K	LR32044	Taiwan
52	Frost King®	ET60FR	906B		U.S.A.
53	National	No. 101, E52811A	362K	LR32044	Taiwan
54	3M Scotch™	Super 33+ on core, 03404NA on packaging	539H/5364	LR48769	U.S.A.
55	Manco®	1219-60	590J	LR31971	Taiwan
56	Victor Automotive Products, Thermoflex	33-UL60, No. 101 E52811A	362K		Taiwan
57	United Tape Company	UT-602	114K/E34833		Taiwan
58	Frost King®	ET60	590J		Taiwan
59	Tuff™ Hand Tools				China
60	Tuff™ Hand Tools				China
61	3M Scotch™	88T			U.S.A.
62	Nitto Denko	No. 228	101K/E34833		Taiwan
63	3M Scotch™	Super 88, 054007-06143	539H	LR48769	U.S.A.
64	3M Scotch™	Super 33+, 10455NA	539H	LR48769	U.S.A.
65	3M Scotch™	700 Commercial Grade, 054007-04218	539H		U.S.A.

Continued.

TABLE 1—*Continued.*

Sample Roll	Brand Name	Product	UL	CSA Reference	Country of Origin
66	L.G. Sourcing, Inc.	19453	206T E62265		Taiwan
67	Manco	P-66	590J	LR31971	Taiwan
68	3M Scotch™	Super 33+	539H	LR48769	U.S.A.
69	3M	Tartan™ 1710, part no. 054007-49656	9Z53		Taiwan
70	Tyco Adhesives National Tape Products	No. 101, E52811A	362K	LR32044	Taiwan
71	Qualpack®	1346, 6-Color			China
72	Nitto Denko	Nitto® No. 228	101K/E34833		Taiwan
73	Frost King®, Thermwell Products Co., Inc.	ET60FR	57RJ		China
74	3M Scotch®	700 Commercial Grade, 054007-04218	539H	LR48769	U.S.A.
75	3M Scotch™	Linerless Electrical Rubber Splicing Tape, 2242, 06165			U.S.A.
76	3M Scotch®	Super 33+, Cold Weather Electrical Tape, 16736NA	539H		U.S.A.
77	3M Scotch®	Super 33+, 054007-06132	539H		U.S.A.
78	3M	Tartan™1710 General Use, 054007-49656	539H/9Z53	LR48769/LR702174	Taiwan
79	3M Scotch®	700 Commercial Grade, 054007-04218	539H	LR48769	U.S.A.
80	3M Scotch®	Super 88, 054007-06143	539H		U.S.A.
81	Ace (Imported for Henkel Capital)	All Weather	362K/E49341	LR32044	Taiwan
82	Ace (Imported for Henkel Capital)	Weather Resistant	362K/E49341	LR32044	Taiwan
83	3M Scotch®	Super 33+, 10414NA	539H		U.S.A.
84	3M	Tartan™ 1710, General Use, 054007-49656	9Z53	LR702174	Taiwan
85	Frost King®, Thermwell Products Co., Inc.	ET60FR	57RJ		China
86	Duck, Henkel Consumer Adhesives	Vinyl Electrical Tape	362K/E49341	LR32044	Taiwan
87	Nitto Denko	No. 21E			China
88	Frost King®, Thermwell Products Co., Inc.	ET60FR	906B		China
89	Power Pro Craft	ETF	VT18/4K71/E220411		China
90	Duck, Henkel Consumer Adhesives	Extra wide electrical tape	74HK/E49341/ATC-F100	232957	China

UL, Underwriters Laboratories.

SEM/EDS

All samples were attached to a pyrolytic carbon planchet using their own adhesives, grounded with carbon paint, and carbon coated by vacuum evaporation. Analysis was performed on a JEOL JSM-6300 SEM with a tungsten filament as the source (JEOL, Peabody, MA). The magnification was roughly 50×, the working distance was approximately 15 mm, the takeoff angle was approximately 30°, and the accelerating voltage was 25 kV. The 4 pi Analysis energy dispersive spectrometer (Durham, NC) was operated with a dead time of approximately 30% and counting time of 200 sec. Although many samples were analyzed once, replicate analyses were conducted on numerous samples to confirm reproducibility of the data.

Evaluation of Discrimination

For each technique, two examiners independently reviewed the data and grouped samples according to similar characteristics/composition. The examiners then compared their groupings and discussed any differences of opinion. If agreement could not be reached, a third examiner reviewed the data and/or the more conservative opinion was taken. For this study, the more conservative opinion was considered to be the one that resulted in less discrimination, which is in direct contrast to the conservative approach that would be taken in casework. In casework, the conservative approach would be to err on the side of discrimination. However, to do so for this study could artificially inflate the discrimination ability. Therefore, when a sample was considered to belong to two separate groups, it was appropriate to merge the two groups. This frequently occurred in relation to width and thickness measurements.

The total number of comparison pairs possible from a population of 90 samples is 4005, calculated with the formula $\frac{n(n-1)}{2}$, where n is the number of samples (10). For each technique, the number of comparison pairs for each indistinguishable group was calculated using the same formula and subsequently summed across the groups to provide the total number of indistinguishable pairs. The percentage of pairs that were discriminated, which is equivalent to the discrimination power (DP), was then calculated as follows:

$$\mathrm{DP} = \%\text{ of pairs discriminated} = 100\% \times \left(1 - \frac{\text{number of indistinguishable pairs}}{\text{total number of comparison pairs}}\right).$$

An example follows for the results of the microscopical examinations. These calculated values were used to compare the relative discrimination of each technique. The discrimination value for the techniques combined was likewise calculated using the indistinguishable sample sets following analysis and comparison of all samples by all techniques.

Results and Discussion

Microscopical Examinations and Physical Examinations

The 90 tape samples were intercompared using the appearance, sheen, width, and thickness of the backing. Figure 1 depicts the sheen/surface features of four different backings. Twenty-four groups resulted, and the first four groups contained 53, 8, 7, and 2 tapes, respectively. The remaining 20 samples were distinguished from all other samples in the collection and therefore formed groups of single samples. These results yielded a discrimination power of 64.3%:

$$DP = 100\% \times \left[1 - \frac{\left(\frac{53(53-1)}{2} + \frac{8(8-1)}{2} + \frac{7(7-1)}{2} + \frac{2(2-1)}{2}\right)}{4005}\right]$$

Table 2 lists the samples by group and includes the widths and thicknesses represented within each group. The largest group included samples with a matte sheen (low gloss) surface, generally had widths between 18.0 and 20.0 mm, and had backing thicknesses ranging from 5.50 to 6.65 mils. The ranges of widths and thicknesses reported for this group were much greater than the 1.0 mm and 0.2 mil tolerances used to differentiate a single pair of samples. This occurrence resulted from numerous evaluations where a sample could not be differentiated from two other samples during the pairwise comparison of their measurements, but the latter two samples could be differentiated from one another. For example, Samples 36 and 39 had average backing thickness measurements of 5.79 and 5.96 mils, respectively; these samples were not differentiated from one another. When the backing thickness of Sample 43 (5.61 mils) was then compared with that of both Samples 36 and 39, it could be differentiated from Sample 39. However, given that both Samples 39 and 43 each form an undifferentiated pair with Sample 36, all three samples were grouped together.

FTIR

FTIR spectra of the backings were examined and segregated based on the presence/absence of peaks as well as ratios of peaks. Grouping of the samples according to similar FTIR spectra resulted in 14 groups, with a resulting discrimination of 83.3%. Table 3 lists the samples represented in each group.

Not only were the groups defined by their spectral patterns, but the chemical compositions of the backings were also evaluated from the resulting spectra and are also included in Table 3. The polymer was the simplest component to classify; three types were identified in this collection: PVC, polyethylene (PE), and butyl rubber (BR). The two black polyethylene-backed tapes were packaged together with other colored tapes, and the package was labeled "Insulated PVC tape." Therefore, it was unexpected that the backings were not PVC, but this should serve as a caution when interpreting product labels. The butyl rubber-backed tape was marketed as an electrical rubber splicing tape, so the butyl rubber composition was not surprising. The presence of some other components within the backings was more difficult to assess owing to peak overlap or presence at low concentrations, but frequently, the presence of an aromatic (likely a phthalate) and/or aliphatic (likely an adipate) component was apparent. Figure 2 is an overlay of the FTIR library spectra for a typical adipate versus a typical phthalate. Figure 3 shows a spectral overlay of two samples that were discriminated by FTIR analysis as a result of the plasticizer content: Sample 42 contains a phthalate as the primary plasticizer whereas Sample 54 contains an adipate. One of the most recognizable differences for evaluating the plasticizer content is that a phthalate has a doublet around 1600 cm^{-1} where not all adipates do. Occasionally, an inorganic component was also noted.

Once the data analysis was completed for the remainder of the analytical techniques, the information obtained via those techniques was used to confirm the infrared classifications. In most instances, the

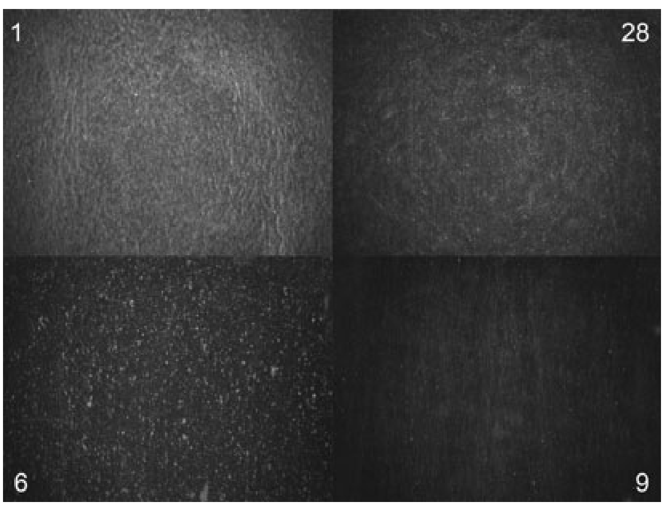

FIG. 1—*Digital micrographs of four different backing surfaces, all taken under the same lighting conditions at a magnification of approximately 30×.*

TABLE 2—*Tape groups as determined by physical characteristics and measurement averages.*

Group	Sample Numbers	Width (mm)*	Backing Thickness (mils)†
A	1, 4, 5, 7, 8, 11, 12, 13, 15, 16, 18, 20, 21, 25, 26, 27, 29, 30, 36, 38, 39, 40, 41, 42, 43, 44, 45, 46, 47, 48, 49, 51, 53, 54, 55, 56, 58, 64, 65, 67, 68, 69, 70, 74, 76, 77, 78, 79, 81, 82, 83, 86, 88	18.0–20.0	5.50–6.65
B	14, 32, 35, 37, 50, 66, 73, 85	18.0–19.0	5.30–6.40
C	10, 17, 23, 24, 61, 63, 80	18.5–19.0	7.30–7.70
D	59, 60	18.0	3.50–4.00
E	2	19.0	4.40
F	3	19.0	4.60
G	6	17.0	4.00
H	9	19.0	6.05
I	19	12.5	6.00
J	22	18.5	6.10
K	28	18.5	5.95
L	31	17.5	4.65
M	33	19.5	4.70
N	34	19.0	5.85
O	52	18.5	5.40
P	57	17.5	6.60
Q	62	19.0	7.20
R	71	15.5	4.20
S	72	19.0	6.50
T	75	19.0	26.30
U	84	18.0	6.45
V	87	18.5	6.05
W	89	19.0	6.20
X	90	37.5	6.35

*Values are rounded to the nearest 0.5 mm.
†Values are rounded to the nearest 0.05 mil.

TABLE 3—*Tape groups as determined by Fourier transform infrared spectroscopy analysis.*

Group	Sample Numbers	Notable Chemical Components
I	33	PVC, adipate
II	17	PVC, adipate, peak at 740 cm^{-1}
III	52	PVC, adipate, peak at 875 cm^{-1}
IV	10, 11, 12, 13, 15, 18, 19, 20, 23, 24, 25, 26, 39, 41, 54, 61, 63, 64, 65, 68, 74, 76, 77, 79, 80, 83	PVC, adipate, some indications of phthalate
V	84	PVC, phthalate, adipate, peak at 875 cm^{-1}
VI	1, 5, 7, 8, 14, 32, 35, 37, 48, 49, 50, 57	PVC, phthalate, adipate, peaks at 875 and 1126 cm^{-1}
VII	6	PVC, phthalate, peak at 875 cm^{-1}
VIII	21, 22, 38, 46, 62, 66, 67, 69, 72	PVC, phthalate, peaks at 1170 cm^{-1} (acrylic?) and 1190 cm^{-1}
IX	2, 3, 4, 9, 31, 42, 45, 51, 53, 55, 56, 58, 70, 71, 81, 82, 86, 87, 89	PVC, phthalate, peaks at 1200 and 1330 cm^{-1}
X	16, 27, 28, 29, 30, 34, 36, 40, 43, 44, 47, 78	PVC, phthalate, peaks at 1165 cm^{-1} (acrylic?), 1200, and 1340 cm^{-1}
XI	85, 88, 90	PVC, phthalate, calcium carbonate, peaks at 712, 1330, and 3640 cm^{-1}
XII	73	PVC, phthalate, calcium carbonate, peaks at 854 and 3640 cm^{-1}
XIII	59, 60	PE
XIV	75	BR, aluminum oxide

PVC, polyvinyl chloride; PE, polyethylene; BR, butyl rubber.

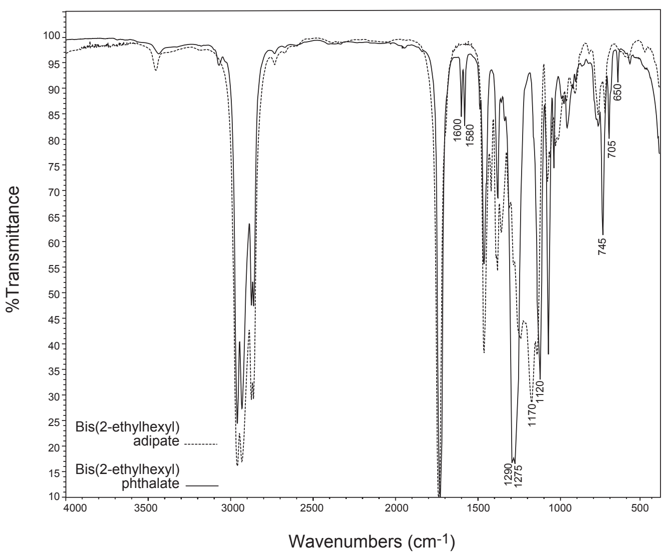

FIG. 2—*Fourier transform infrared spectroscopy spectral overlay of a common adipate plasticizer and a common phthalate plasticizer. Major peaks differences are labeled. Library spectra were reproduced from the HR Polymer Additives and Plasticizers library, Thermo Fisher Scientific, Inc., for Nicolet FT-IR.*

general classifications were confirmed. However, Py-GC/MS analysis did not confirm the presence of a phthalate in some of the samples in Group IV. Further, the possible acrylic noted in the samples from Groups VIII and X was not confirmed. Regarding the inorganic components noted through FTIR analysis, indications of each of these were demonstrated by elemental analysis using SEM/EDS.

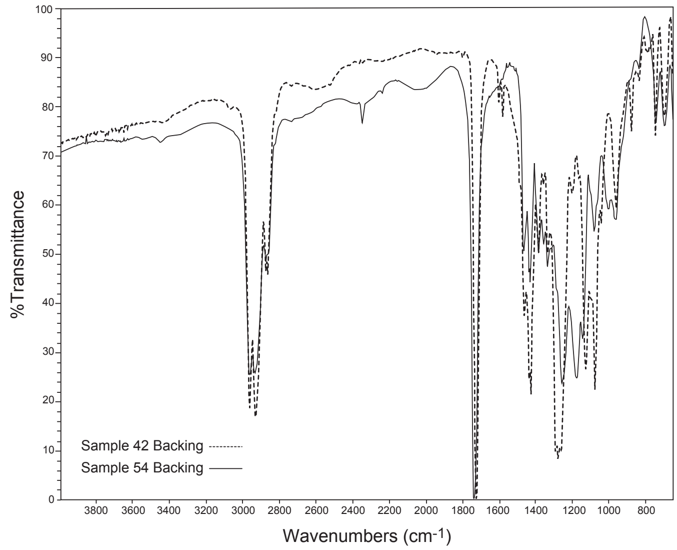

FIG. 3—*Fourier transform infrared spectroscopy spectral overlay of two backings that differ: Sample 42 contains a phthalate and Sample 54 contains an adipate.*

TABLE 4—*Tape groups as determined by pyrolysis–gas chromatography/mass spectrometry analysis.*

Group	Sample Numbers	Notable Chemical Components
a	2, 3, 4, 6, 8, 9, 21, 22, 31, 32, 38, 42, 45, 46, 51, 53, 55, 56, 58, 66, 67, 70, 71, 81, 82, 86, 87, 88, 89, 90	PVC, phthalate
b	16, 27, 28, 29, 30, 34, 36, 40, 43, 44, 47, 78	PVC, mixture of phthalates
c	14, 35, 37, 50, 73, 85	PVC, phthalate, mixture of phthalates
d	1, 5, 7, 48, 49, 57, 62, 69, 72	PVC, phthalate, trimellitate
e	33	PVC, mixture of phthalates, methyl methacrylate, adipate, small amount of a sebacate
f	10, 11, 12, 13, 15, 18, 19, 20, 23, 24, 25, 26, 39, 41, 54, 61, 63, 64, 65, 68	PVC, adipate, sebacate
g	74, 76, 77, 79, 80, 83	PVC, adipate, mixture of phthalates, sebacate
h	84	PVC, two adipates, mixture of phthalates, sebacate
i	17	PVC, two adipates, azelaic acid plasticizer
j	52	PVC, mixture of adipates, phthalate, possible glutarate
k	59, 60	PE
l	75	BR

PVC, polyvinyl chloride; PE, polyethylene; BR, butyl rubber.

Py-GC/MS

Evaluation of the Py-GC/MS data separated the 90 tapes into 12 groups, with a resulting discrimination ability of 81.0%. As with

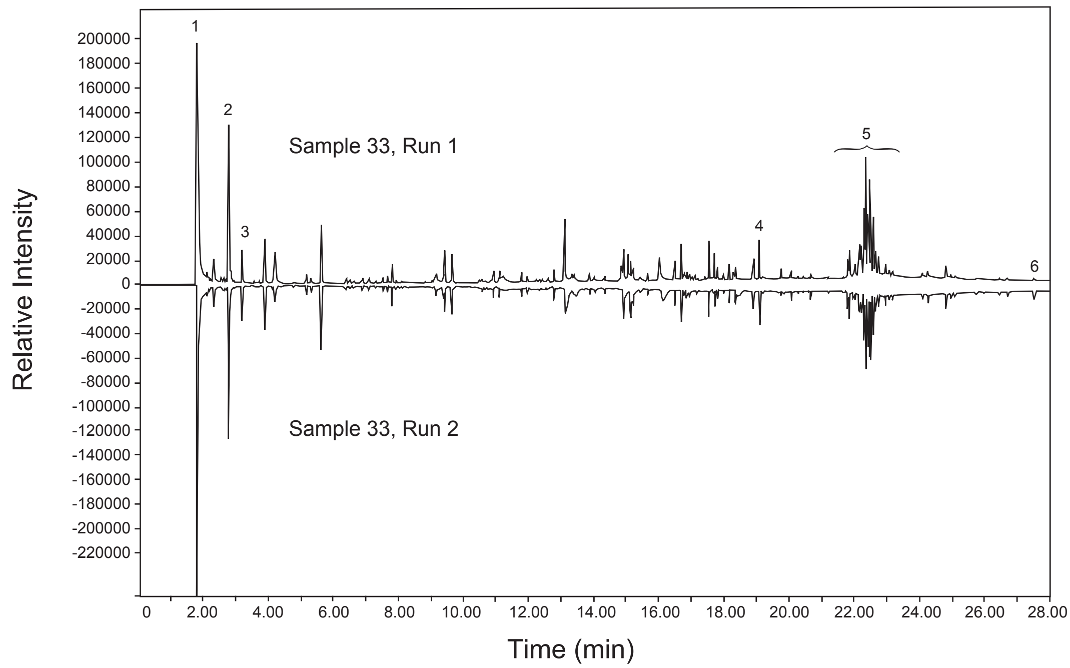

FIG. 4—*Pyrolysis–gas chromatography/mass spectrometry chromatograms of replicates of Sample 33 collected by two different analysts on two different days. Peaks are as follows: (1) hydrogen chloride, (2) benzene, (3) methyl methacrylate, (4) an adipate plasticizer, (5) a mixture of phthalate plasticizers, and (6) a sebacate plasticizer.*

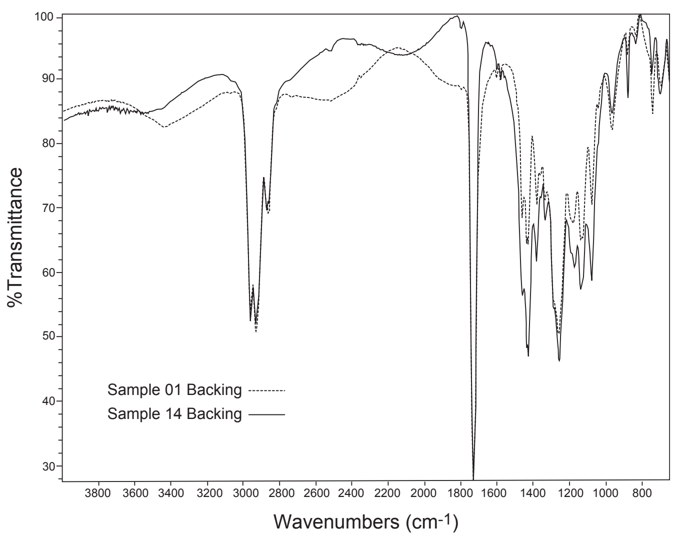

FIG. 5—*Fourier transform infrared spectroscopy spectral overlay of two backings (Samples 1 and 14) that were not discriminated by this technique.*

the FTIR data, the Py-GC/MS data were used to evaluate the range of chemical compositions found in the backings of electrical tapes. However, much more information, specifically regarding the plasticizer content, was able to be elucidated from the Py-GC/MS data. Table 4 shows the breakdown of these groups with their primary chemical compositions included.

The presence of PVC is indicated by hydrogen chloride and a series of aromatics (benzene, naphthalene). Several classes of plasticizers were observed, including phthalates, adipates, sebacates, azelates, and trimellitates. The phthalates included either bis(2-ethylhexyl) phthalate or dioctyl phthalate. Likewise, the adipates, sebacates, and azelates included either the bis(2-ethylhexyl) or the dioctyl isomers of each class. All of these plasticizer components eluted from the GC column significantly later than the PVC components. Further, methyl methacrylate was noted in one sample (Sample 33), and this peak eluted among the PVC aromatic components. Figure 4 indicates the notable chemical components of Sample 33, and it also demonstrates the typical reproducibility achieved at the authors' laboratory for the technique. These pyrograms represent analyses by two different analysts on separate days.

Three non-PVC-based samples were found, two of which were polyethylene and one was butyl rubber. The polyethylene pyrograms exhibit the typical fragmentation pattern for that material, consisting of a series of diene, alkene, and alkane hydrocarbon chains. The butyl rubber pyrogram contains dipentene and isobutene oligomers.

Samples 1 and 14 were too similar to conclusively distinguish by FTIR but could be differentiated by Py-GC/MS, as Sample 1 contained a trimellitate that Sample 14 did not. Their resulting spectra and pyrograms are depicted in Figs 5 and 6.

FTIR was unable to provide additional discrimination over Py-GC/MS in the analysis of the adhesives (3). However, FTIR did provide additional information in the analysis of the backings; Figs 7 and 8 show the spectra and pyrograms of one such pair in which FTIR was able to discriminate samples that Py-GC/MS could not.

SEM/EDS

The presence or absence of PVC and various flame retardants and additives in the electrical tape backings allowed for considerable discrimination using SEM/EDS. Four major defining elemental characteristics were observed, most of which could be further broken down into subsets for additional discrimination. The four defining characteristics were the following: (1) intense chlorine with aluminum and silicon both present in significant amounts, (2)

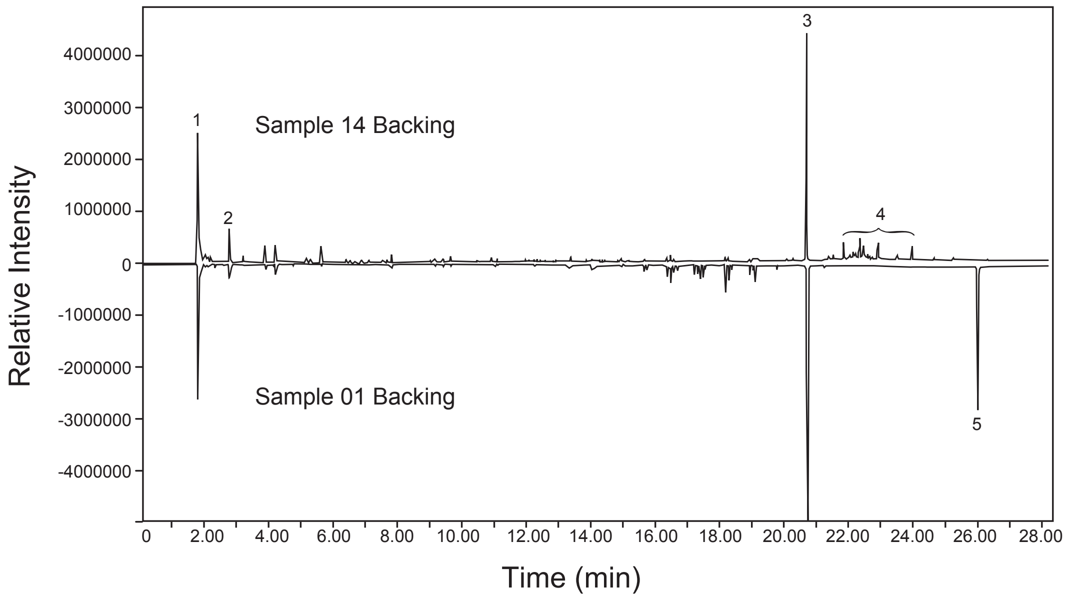

FIG. 6—*Pyrolysis–gas chromatography/mass spectrometry chromatograms of Samples 1 and 14, demonstrating they differ. Peaks are as follows: (1) hydrogen chloride, (2) benzene, (3) a phthalate plasticizer, (4) a mixture of phthalate plasticizers, and (5) a trimellitate plasticizer.*

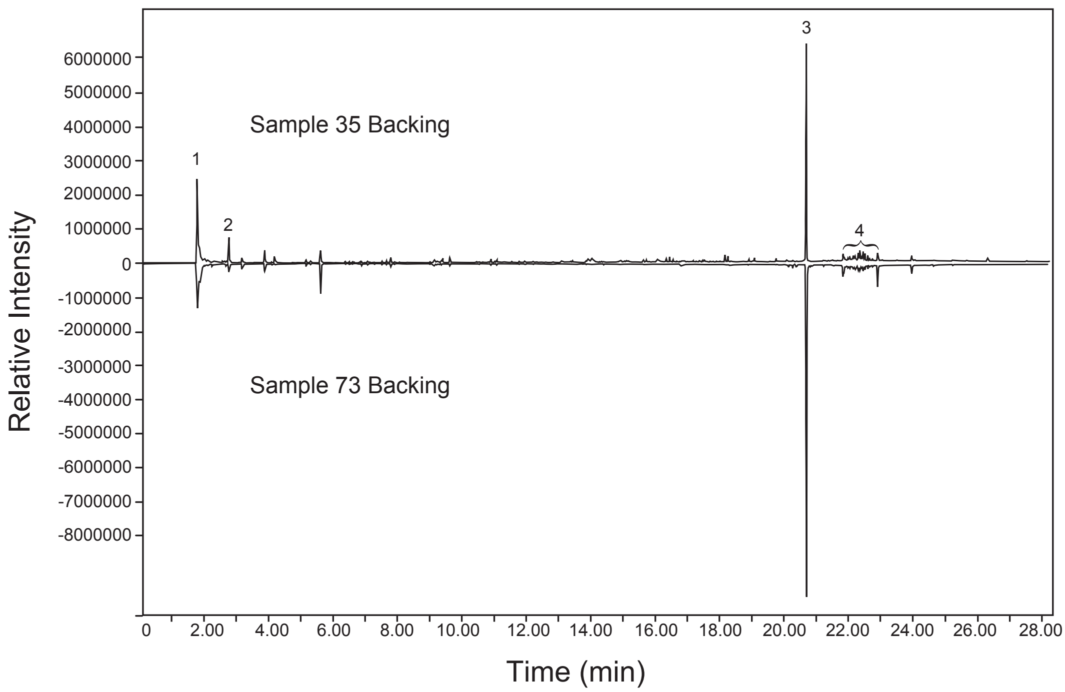

FIG. 7—*Pyrolysis–gas chromatography/mass spectrometry chromatograms of two backings (Samples 35 and 73) that were not discriminated by this technique. Peaks are as follows: (1) hydrogen chloride, (2) benzene, (3) a phthalate plasticizer, and (4) a mixture of plasticizers.*

intense chlorine with calcium and/or antimony present in significant amounts, (3) chlorine only, and (4) minimal, if any, chlorine. Representative spectra for each of these four defining characteristics are depicted in Fig. 9. Within the samples with significant aluminum and silicon, additional discrimination was obtained based on the presence or absence of calcium, titanium, and antimony. Figure 10 shows how these samples separated into Groups i through iv. In the end, all samples were divided into 15 groups with a resulting discrimination of 87.3%. Table 5 outlines the various groups noted.

Spectral interpretation was not always straightforward. As expected with SEM/EDS, peak overlap can make spectral interpretation difficult. Of particular note for electrical tape backings, specifically for the SEM/EDS Groups vii through x, overlap of the antimony and calcium peaks can easily lead to misinterpretation of the presence of tin. Tin may or may not be present in these samples in small amounts, but the presence/amounts of calcium and/or antimony peaks prevented tin from being confirmed.

Comparing the SEM/EDS results with those of FTIR, SEM/EDS provided a much greater degree of elemental

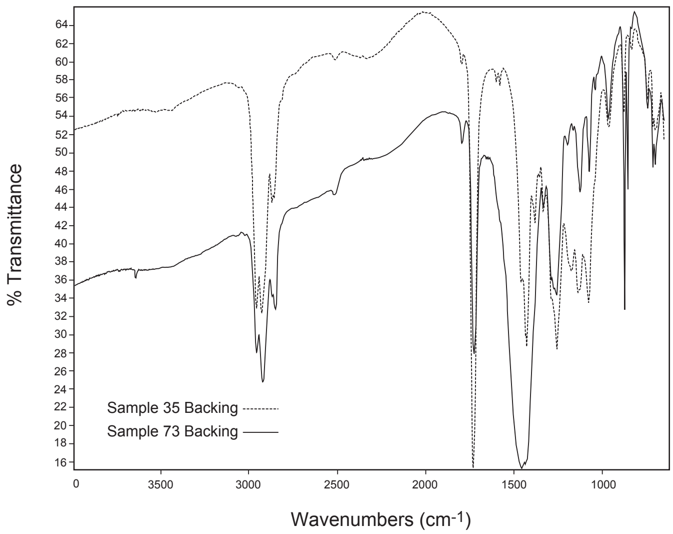

FIG. 8—*Fourier transform infrared spectroscopy spectral overlay of Samples 35 and 73, demonstrating they differ.*

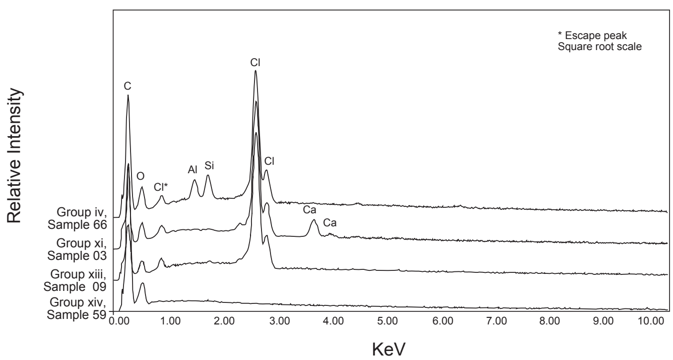

FIG. 9—*Scanning electron microscopy/energy-dispersive spectroscopy spectral overlay (displayed in square root scale) of samples representing the four major defining classes of elemental characteristics.*

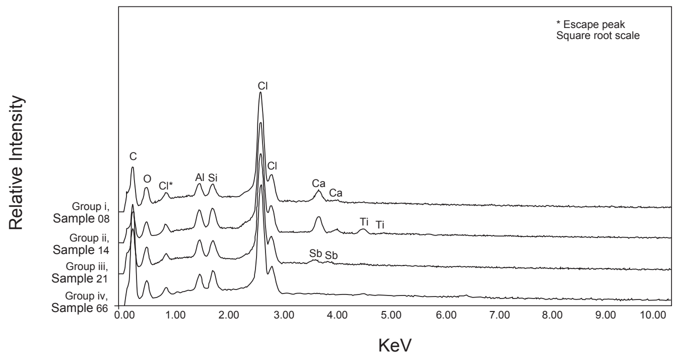

FIG. 10—*Scanning electron microscopy/energy-dispersive spectroscopy spectral overlay (displayed in square root scale) of representative samples of the four groups that contain significant amounts of aluminum and silicon.*

information. For example, not all samples containing calcium had indications of calcium carbonate in the IR spectrum. However, as mentioned previously, when an inorganic component was noted in an FTIR spectrum, indications of it were also present in the EDS spectrum (e.g., calcium carbonate by FTIR, calcium by SEM/EDS, or, aluminum oxide by FTIR, aluminum by SEM/EDS).

Techniques Combined

Following the evaluation of the individual techniques, the discrimination of the combined techniques was assessed. The result was that 94.3% of the tapes could be discriminated following backing analysis via the full analytical protocol carried out in this study. Table 6 displays the sample groupings for the backings according to indistinguishable physical characteristics and chemical compositions, along with the available product information.

Adhesive and Backing Results Combined

Incorporating the previously published results for the adhesives with these current results for the backings, additional discrimination was achieved. The groups of indistinguishable backings that were further discriminated based on their adhesives were G9, G30, and

TABLE 5—*Tape groups as determined by scanning electron microscopy/energy-dispersive spectroscopy analysis.*

Group	Defining Elemental Characteristics	Sample Numbers	Notable Elements Present
i	Intense Chlorine with Aluminum and Silicon both present in significant amounts	4, 8, 32, 42, 45, 51, 52, 53, 55, 56, 58, 70, 81, 82, 86	Cl, Al, Si, Ca
ii		14, 35, 37, 50	Cl, Al, Si, Ca, Ti
iii		21, 38, 46, 67	Cl, Al, Si, Sb
iv		66	Cl, Al, Si
v	Intense Chlorine with Calcium and/or Antimony present in significant amounts	22, 69	Cl, Sb
vi		72, 74, 76, 77, 79, 80, 83	Cl, Sb, Al, Mg
vii		62	Cl, Sb > Ca, Al, Si, Ti
viii		2, 10, 11, 12, 13, 15, 17, 18, 19, 20, 23, 24, 25, 26, 27, 28, 39, 41, 54, 61, 63, 64, 65, 68	Cl, Sb > Ca, Pb
ix		16, 29, 30, 34, 36, 40, 43, 44, 47	Cl, Ca > Sb, Pb
x		1, 5, 7, 48, 49, 57, 78, 84	Cl, Ca > Sb
xi		3, 6, 31, 71, 87, 88, 89, 90	Cl, Ca
xii		73, 85	Cl, Ca, Ti
xiii	Chlorine only	9, 33	Cl
xiv	Minimal, if any, chlorine	59, 60	n/a
xv		75	Al

TABLE 6—*Tape groups following all backing examinations.*

Group Number	Sample Roll	Brand Name	Product	UL	CSA Reference	Country of Origin
G1	1	Marcy Enterprises, Inc.	MA 750	111K		Taiwan
	5	Tape It, Inc.	E-60	119K		Taiwan
	7	Marcy Enterprises, Inc.	MA 750	111K		Taiwan
	48	Tape-It	36-T			U.S.A.
	49	Tape-It	36-T			U.S.A.
G2	2	Advance®	AT7, BS3924, 31/90Tp			England
G3	3	Work Saver™, a Royal Tools brand	Stock no. 55, 5 color P.V.C Tape Assortment			China
G4	52	Frost King®	ET60FR	906B		U.S.A.
G5	73	Frost King®, Thermwell Products Co., Inc.	ET60FR	57RJ		China
G6	85	Frost King®, Thermwell Products Co., Inc.	ET60FR	57RJ		China
G7	88	Frost King®, Thermwell Products Co., Inc.	ET60FR	906B		China
G8	14	Frost King®	ET60	206T		Taiwan
	35	Frost King®	ET60	206T		Taiwan
	37	National	All-Purpose Grade	206T		Taiwan
	50	General Electric	GE2472-31D	206T		Taiwan
G9	4	Tesa Tape, Inc.	40201, No. 111 E52811A	362K		Taiwan
	42	National	All-Purpose	362K		Taiwan
	45	Calterm®	49605	590J		Taiwan
	51	National	No. 101, E52811A	362K	LR32044	Taiwan
	53	National	No. 101, E52811A	362K	LR32044	Taiwan
	55	Manco®	1219-60	590J	LR31971	Taiwan
	56	Victor Automotive Products, Thermoflex	33-UL60, No. 101 E52811A	362K		Taiwan
	58	Frost King®	ET60	590J		Taiwan
	70	Tyco Adhesives, National Tape Products	No. 101, E52811A	362K	LR32044	Taiwan
	81	Ace (Imported for Henkel Capital)	All Weather	362K/E49341	LR32044	Taiwan
	82	Ace (Imported for Henkel Capital)	Weather Resistant	362K/E49341	LR32044	Taiwan
	86	Duck, Henkel Consumer Adhesives	Vinyl Electrical Tape	362K/E49341	LR32044	Taiwan
G10	90	Duck, Henkel Consumer Adhesives	Extra wide electrical tape	74HK/E49341/ATC-F100	232957	China
G11	8	Manco®	200 MPH, AE-66	590J	LR31971	Taiwan
G12	21	Manco®	P-66	590J	LR31971	Taiwan
	38	Manco®	P-660	590J	LR31971	Taiwan
	46	Manco®	P-20	590J	LR31971	Taiwan
	67	Manco	P-66	590J	LR31971	Taiwan
G13	22	Manco®	667 Pro Series™	590J	LR31971	Taiwan
G14	6	Qualpack®	1346, 6-Color			China
G15	71	Qualpack®	1346, 6-Color			China
G16	9	Archer® (Packaged for Radio Shack)	64-2349	590J		Taiwan
G17	31	Regal®	Model ET-6			Taiwan
G18	57	United Tape Company	UT-602	114K/E34833		Taiwan
G19	59	Tuff™ Hand Tools				China
	60	Tuff™ Hand Tools				China
G20	66	L.G. Sourcing, Inc.	19453	206T/E62265		Taiwan
G21	62	Nitto Denko	No. 228	101K/E34833		Taiwan
G22	72	Nitto Denko	Nitto® No. 228	101K/E34833		Taiwan

Continued.

TABLE 6—*Continued.*

Group Number	Sample Roll	Brand Name	Product	UL	CSA Reference	Country of Origin
G23	87	Nitto Denko	No. 21E			China
G24	89	Power Pro Craft	ETF	VT18/4K71/E220411		China
G25	32	GE	GE2472-3DD	206T		Taiwan
G26	10	3M Scotch™	Super 88, 054007-06143	539H	LR48769	U.S.A.
	23	3M Scotch™	Super 88, 054007-06143	539H	LR48769	U.S.A.
	24	3M Scotch™	Super 88, 054007-06143	539H	LR48769	U.S.A.
	61	3M Scotch™	88T			U.S.A.
	63	3M Scotch™	Super 88, 054007-06143	539H	LR48769	U.S.A.
G27	17	3M Scotch™	Super 88 054007-06143	539H	LR48769	U.S.A.
G28	80	3M Scotch®	Super 88, 054007-06143	539H		U.S.A.
G29	19	3M Scotch™	Super 33+, Cat. 194NA	539H	LR48769	U.S.A.
G30	11	3M Scotch™	Super 33+, 10414 NA	539H	LR48769	U.S.A.
	12	3M Scotch™	Super 33+, 10455 NA	539H	LR48769	U.S.A.
	13	3M Scotch™	Super 33+	539H	LR48769	U.S.A.
	15	3M Scotch™	Super 33+, 10455 NA	539H	LR48769	U.S.A.
	18	3M Scotch™	Super 33+, Cat. 195NA	539H	LR48769	U.S.A.
	20	3M Scotch™	Super 33+, 10414 NA	539H	LR48769	U.S.A.
	25	3M Scotch™	Super 33+, 054007-06132	539H	LR48769	U.S.A.
	26	3M Scotch™	Super 33+, 054007-06132	539H	LR48769	U.S.A.
	39	3M Scotch™	Super 33+, 3744NA	539H	LR48769	U.S.A.
	41	3M Scotch™	Super 33+, 200NA	539H	LR48769	U.S.A.
	54	3M Scotch™	Super 33+ on core, 03404NA on packaging	539H/5364	LR48769	U.S.A.
	64	3M Scotch™	Super 33+, 10455NA	539H	LR48769	U.S.A.
	65	3M Scotch™	700 Commercial Grade, 054007-04218	539H		U.S.A.
	68	3M Scotch™	Super 33+	539H	LR48769	U.S.A.
G31	74	3M Scotch®	700 Commercial Grade, 054007-04218	539H	LR48769	U.S.A.
	76	3M Scotch®	Super 33+, Cold Weather Electrical Tape, 16736NA	539H		U.S.A.
	77	3M Scotch®	Super 33+, 054007-06132	539H		U.S.A.
	79	3M Scotch®	700 Commercial Grade, 054007-04218	539H	LR48769	U.S.A.
	83	3M Scotch®	Super 33+, 10414NA	539H		U.S.A.
G32	16	3M	Tartan™ 1710, part no. 054007 49656	539H	LR48769	U.S.A.
	29	3M	Temflex™, 1700, 54007-69764	539H	LR48769	U.S.A.
	30	3M	Temflex™, 1700, 54007-69764	539H	LR48769	U.S.A.
	36	3M	Tartan™ 1710, part no. 49656	539H		U.S.A.
	40	3M	Tartan™ 1710, part no. 054007 49656	539H	LR48769	U.S.A.
	43	3M	Tartan™ 1710, part no. 054007 49656	539H	LR48769	U.S.A.
	44	3M	Tartan™ 1710, part no. 054007 49656	539H	LR48769	U.S.A.
	47	3M	Tartan™ 1710, part no. 054007 49656	539H	LR48769	U.S.A.
G33	27	3M	Tartan™ 1710, part no. 054007 49656	539H	LR48769	U.S.A.
G34	28	3M	Tartan™ 1710, part no. 054007 49656	539H	LR48769	U.S.A.
G35	34	3M	Tartan™ 1710, part no. 54007-49656	539H	LR48769	U.S.A.
G36	69	3M	Tartan™ 1710, part no. 054007-49656	9Z53		Taiwan
G37	78	3M	Tartan™1710 General Use, 054007-49656	539H/9Z53	LR48769/LR702174	Taiwan
G38	75	3M Scotch™	Linerless Electrical Rubber Splicing Tape, 2242, 06165			U.S.A.
G39	84	3M	Tartan™ 1710, General Use, 054007-49656	9Z53	LR702174	Taiwan
G40	33	3M Scotch™	Cat. 190			U.S.A.

UL, Underwriters Laboratories.

G31. Within G9, Sample 4 was further differentiated from the others. Additionally, Samples 42, 51, 53, and 56 formed a subgroup as did Samples 45, 55, 58, 70, 81, 82, and 86. For G30, Sample 65 was further differentiated from the other samples. G31 was divided into two subgroups, the first of which included Samples 74 and 79; the second included Samples 76, 77, and 83.

TABLE 7—*Relative discrimination power of the techniques.*

Technique	Discrimination of Adhesives (%)	Discrimination of Backings (%)
Stereomicroscopy and Physical Measurements	53	64
FTIR	67	83
Py-GC/MS	83	81
SEM/EDS	17	87
Techniques Combined	85	94
Adhesives and Backings Combined	96	

FTIR, Fourier transform infrared spectroscopy; Py-GC/MS, pyrolysis–gas chromatography/mass spectrometry; SEM/EDS, scanning electron microscopy/energy-dispersive spectroscopy.

It is rare that electrical tape samples are received in casework in pristine form. Often, the adhesive layer is contaminated by use or environmental exposure. These factors can complicate assessment of overall thickness measurements. Moreover, electrical tape adhesives are very thin relative to the backing and, as such, are a minor contributor to the overall thickness. For these reasons, only backing thicknesses were considered as points of comparison in the thickness measurements reported in this study. In actual casework, overall thickness measurements are assessed when the condition of the samples allows.

The overall discrimination achieved with the intact tape samples reached 95.76%. As there were potentially duplicate samples of the same products and the sample set was not meant to be representative of electrical tape available in any particular geographical location, caution is advised on drawing conclusions as to how these discrimination values can be applied to comparisons of casework samples.

Table 7 displays the relative discrimination power of each technique for the backings as well as for the adhesives. These values provide a means of evaluating and comparing the discrimination power of various techniques for the analysis of electrical tape components. With the exception of Py-GC/MS, the discrimination for each technique was higher for the backings than for the adhesives. It is useful to know the difference in discrimination power between the backings and the intact tape specimen in the event that the adhesive of an evidentiary tape specimen is too degraded or contaminated to properly characterize.

These discrimination values can also be weighed in conjunction with the additional advantages and disadvantages of the techniques (e.g., ease of use and availability) to develop an analytical scheme for the analysis of electrical tapes. Further, it provides a baseline to assess how feasible alternative techniques (e.g., isotope-ratio mass spectrometry [11]) are for the analysis of these materials.

Protocol Recommendations

Based on the results of this study, the FBI Laboratory does not plan to modify its standard procedure for electrical tape analysis. Compared with Py-GC/MS and SEM/EDS, FTIR is the simplest, quickest, and most widely available technique, making it the logical first choice for instrumental analysis of electrical tape backings. SEM/EDS was the most discriminating technique for the backings, but it is more labor-intensive and requires more skill. Even the least discriminating instrumental technique for the backings, Py-GC/MS, did provide additional discrimination; however, it is destructive to the sample. In most instances, therefore, SEM/EDS will continue to be used prior to Py-GC/MS within the authors' laboratory.

The FBI Laboratory does not include sourcing of electrical tapes of unknown origin in its protocol, nor does it plan to as a result of this study. As previously mentioned, the sample set was not meant to be used for sourcing purposes, and caution is advised in making assumptions based on any apparent correlations observed within the reported results.

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Additional information and reprint requests:
Andria Hobbs Mehltretter, M.S.
Laboratory Division
Federal Bureau of Investigation
2501 Investigation Parkway
Room 4220
Quantico, VA 22135
E-mail: andria.mehltretter@ic.fbi.gov